Defense Technical Information Center Compilation Part Notice

ADP014336

TITLE: A Small Angle Polarized Neutron Scattering Investigation of Magnetic Correlations in Nanocrystalline Fe89Zr7B3Cu1

This paper is part of the following report:

TITLE: Materials Research Society Symposium Proceedings. Volume 746. Magnetoelectronics and Magnetic Materials - Novel Phenomena and Advanced Characterization

Mat. Res. Soc. Symp. Proc. Vol. 746 © 2003 Materials Research Society R3.9

A Small Angle Polarized Neutron Scattering Investigation of Magnetic Correlations in Nanocrystalline $Fe_{89}Zr_7B_3Cu_1$

Enrico Agosti, Oscar Moze, John Cadogan[1], Kiyonori Suzuki[1*], Andre Heinemann[2] and Armen Hoell[2]
INFM - S[3], Dipartimento di Fisica, Università degli Studi di Modena e Reggio Emilia, Italy.
[1]School of Physics, University of New South Wales, Sydney, Australia.
[2]Berlin Neutron Scattering Centre, Hahn Meitner Institut, Berlin, Germany.
*now at The School of Physics and Materials Engineering, Monash University, Australia

ABSTRACT

The technique of small angle neutron scattering (SANS) is ideally suited for determining the length scale of magnetic correlations in nanocrystalline materials. The additional use of polarized neutrons also allows for a clear separation between magnetic and non-magnetic scattering. The temperature dependence of the SANS cross-section from a two-phase alloy, consisting of both amorphous and nano-crystalline parts, $Fe_{89}Zr_7B_3Cu_1$, has been measured in the temperature range from 293 to 500 K. The SANS measurements are accompanied by bulk magnetization and Mössbauer transmission data. In this range of temperatures, the magnetic contrast between the nanocrystalline and amorphous phases, which are both magnetic, changes dramatically. This phase contrast increases up to 380 K, which is the proposed decoupling temperature for the inter-granular exchange stiffness. Above this temperature, the contrast levels off slowly, being totally dominated by the decreasing magnetization of the nanocrystalline phase.

INTRODUCTION

Magnetic softness of exceptional quality is known to exist in both structurally amorphous and nanocrystalline systems. Two-phase Fe based nanocrystalline materials which consist of bcc nanocrystallites and a residual amorphous magnetic matrix are also excellent soft magnetic materials. The corresponding anisotropy energy of amorphous systems is based on the Random Anisotropy Model [1], a model which even accounts for the anisotropy in more structured nano-crystalline systems [2]. An extended model proposes a penetration of the Fe exchange field from the nanocrystal phase into the amorphous phase and partially describes the magnetic hardening present in such materials [3]. The dramatic increase with temperature near the Curie point of the amorphous phase has been accounted for in the framework of an extended two-phase Random Anisotropy Model [4]. The coercivity H_c is predicted, in the vicinity of the Curie point of the amorphous phase T_c^{am}, to vary as the -6^{th} power of the spontaneous magnetization of the amorphous phase. Above T_c^{am}, H_c is also very sensitive to the volume of the amorphous phase, V_{am} [5].

Small angle neutron scattering (SANS) allows density and magnetization fluctuations to be investigated on the length scales which occur in nanostructured magnets such as FINEMET [6]. In contrast to conventional SANS, use of polarized neutrons allows for a clear separation of magnetic from nuclear scattering [7]. Reported here is a direct determination of the temperature

dependence of the contrast between the magnetic nanocrystalline and amorphous phases in $Fe_{89}Zr_7B_3Cu_1$ by use of polarized SANS. The data show clear and unambiguous microscopic evidence, at a nanoscale level, for the magnetic decoupling between the two phases.

EXPERIMENTAL DETAILS

A sample of $Fe_{89}Zr_7B_3Cu_1$ was prepared in an argon atmosphere by a single roller melt spinner, sealed in evacuated quartz tubes (at a pressure of 10^{-3} Pa) and annealed for one hour at 745 K using a salt bath. The average grain size of the nanocrystalline particles, 12 nm, was determined by X-ray diffraction from the peak width of the bcc-(110) reflection. ^{57}Fe Mössbauer spectra were obtained using a conventional constant-acceleration spectrometer and a ^{57}Co Rh source. Coercivity was measured using a DC *B-H* loop tracer in the temperature range from 120 to 473 K. The volume fraction of the amorphous phase, approx. 60%, was evaluated from the Mössbauer spectra. Polarized Small Angle Neutron Scattering measurements were performed on the V4 instrument, located at the BENSC BERII reactor, Hahn Meitner Institut, Berlin, Germany [8].

RESULTS AND DISCUSSION

The temperature dependence of H_c for $Fe_{89}Zr_7B_3Cu_1$ is displayed in figure 1. A decoupling between the two phases at a temperature of $T_c^{am} = 380$ K is inferred by assuming that the amorphous exchange interaction is Heisenberg like, with an associated critical exponent, $\beta = 0.36$ for the magnetization. In this context, the extended RMA model predicts that the temperature dependence of H_c varies as $(T_c^{am} - T)^{-6\beta}$ [4]. This variation of H_c is also displayed in figure 1.

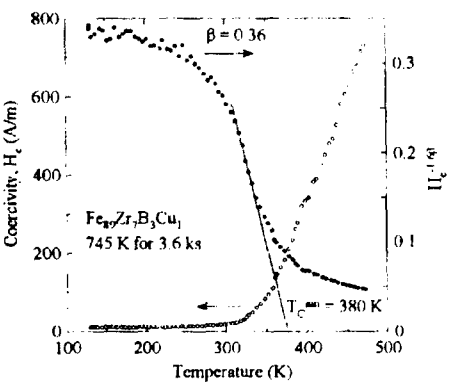

Figure 1. Temperature dependence of the coercivity field H_c for $Fe_{89}Zr_7B_3Cu_1$

A further estimate of the Curie point of the amorphous phase has been inferred by a least squares fitting analysis of the temperature dependence of the mean hyperfine field B_{hf} for the amorphous phase, obtained from the Mossbauer spectra, using the model of Handrich [9]. This is displayed in figure 2. Even though this method appears to underestimate the Curie point determined by the previous technique, lack of Mossbauer data above 300 K may account for this discrepancy.

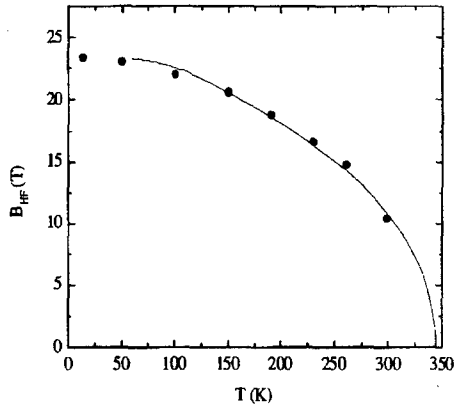

Figure 2. Temperature dependence of the measured mean hyperfine field B_{hf} for the amorphous phase of $Fe_{89}Zr_7B_3Cu_1$ (•) and the fitted dependence using the Handrich model (—).

In the limit of scattering at small angles, the neutron scattering amplitude for a particles i (nuclear or magnetic) of volume V_i, located at positions r_i, embedded in a homogeneuous amorphous matrix is given by equation 1;

$$F(q) = \int dr\Delta\eta_i \exp(iq.r_i) = \Delta\eta_i V_i f(qR) \qquad (1)$$

where $\Delta\eta_i = \Delta\eta_{particle} - \Delta\eta_{matrix}$ is the amplitude contrast between particle and matrix scattering densities, $\vec{q}$ is the neutron wave-vector transfer and f(qR) is the particle form factor, a function which depends only on the shape of the particle with a nominal size R. For a system with a low fraction of independent scattering particles, the small angle scattering probability I(q), when summed over all scattering centres, is directly proportional to the square of the scattering amplitude, as given by equation 2;

$$I(q) = F(q)^2 \qquad (2)$$

The use of polarized neutron in a SANS experiment allows the separate *nuclear* and *magnetic* contributions to the scattering amplitude to be evaluated with more accuracy than by

use of conventional SANS [7]. The q-dependence of the *magnetic* contribution to I(q) at 330 K is displayed below in figure. 3.

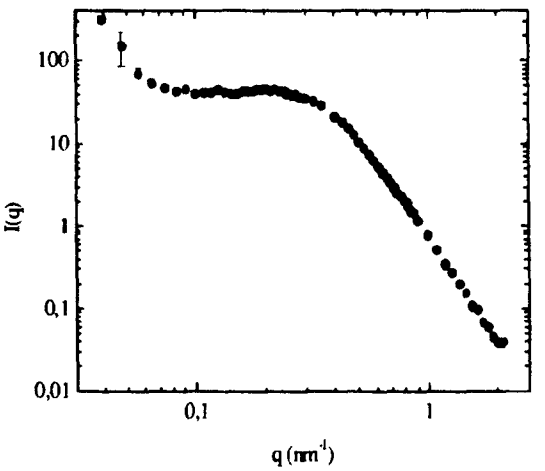

Figure 3. The q-dependence of the magnetic contribution to I(q) for $Fe_{89}Zr_7B_3Cu_1$ at 330 K.

An extremely relevant aspect of the SANS technique is the relationship between the contrast $\Delta M = M_{am} - M_{nano}$ of the magnetic parts of the amorphous and nanocrystal phases and the magnetic scattering probability I(q) . This is given by the invariant quantity [10]:

$$c_p c_{am} V|\Delta M|^2 = 2\pi^2 \int_0^\infty q^2 I(q) dq \qquad (3)$$

where c_p and c_{am} are the concentrations of particle and amorphous phases, respectively, for a sample volume V. The temperature dependence of the magnetic contrast has been determined in the range from 290 to 475 K. This is displayed in figure 4, together with the nuclear contrast, which has also been determined using equation (3) via the measured nuclear scattering probability. The error bars associated with the nuclear scattering are larger than their magnetic counterparts as the magnetic scattering is significantly larger than its nuclear counterpart. Use of conventional SANS would, however, preclude such a clear separation between the two types of scattering. As expected, the nuclear contrast displays no temperature dependence, whilst in the same temperature interval, the magnetic contrast increases up to 380 K, which is the proposed decoupling temperature for the inter-granular exchange stiffness. Above this temperature, the contrast levels off slowly as it is totally dominated by the slowly decreasing magnetization of the

nanocrystalline phase, M_{nano}. The behaviour of the magnetic contrast is very clear and unambiguous microscopic evidence for the decoupling.

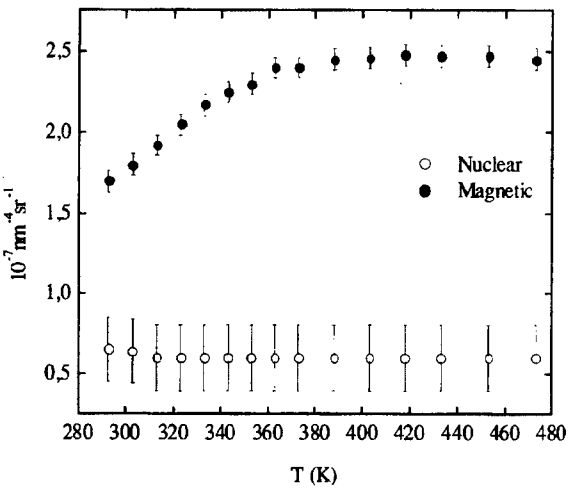

Figure 4. Temperature dependence of the magnetic and nuclear contrasts for $Fe_{89}Zr_7B_3Cu_1$.

The volume packing fraction of the Fe based nanocrystals, at approximately 40% is rather high in the system under investigation. This introduces significant interference effects between particles, a situation that is difficult to treat analytically [11]. However, SANS measurements are planned on samples with a much lower concentration of nanocrystalline grains in order to characterize in detail the length scale (or correlation length) over which magnetic fluctuations exist. What is clear from the present investigation is that the use of polarized SANS is a sensitive and unique structural probe for characterizing fluctuations in atomic and magnetic density in two-phase nanostructured magnets.

CONCLUSIONS

The technique of small angle neutron scattering (SANS) is ideally suited for determining the length scale of magnetic correlations in nanocrystalline materials. Polarized small angle scattering allows for a clear separation between magnetic and non-magnetic scattering in two-phase nanostructured magnets. The technique has been utilized to investigate the temperature dependence of the microscopic magnetic contrast between amorphous and nano-crystalline parts in $Fe_{89}Zr_7B_3Cu_1$, in the temperature range from 293 to 500 K. The decoupling temperature for the inter-granular exchange stiffness has been directly observed and compares well with that inferred from indirect bulk measurements.

ACKNOWLEDMENTS

Research supported by the INFM Research Center – S^3 (Nanostructures and Biosystems at Surfaces), University of Modena and Reggio Emilia, Italy and the Australian Research Council. This work was also carried out in the framework of the European Human Capital and Mobility Programme.

REFERENCES

1. R. Alben, J. J. Becker, and M. C. Chi, *J. Appl. Phys.* **49**, 1653 (1978).
2. G. Herzer, *IEEE Trans. Magn.* **25**, 3327 (1989).
3. A. Hernando, M. Vazquez, T. Kulik and C. Prados, *Phys. Rev. B*, **51**, 3581 (1995).
4. K. Suzuki and J. M. Cadogan, *Phys. Rev. B* **58**, 2730 (1998).
5. K. Suzuki and J. M. Cadogan, *J. Magn. Mag. Matls.* **203**, 229 (1999).
6. M. Ohnuma, J. Suzuki, S. Funahashi, T. Ishigaki, H. Kuwano, Y. Hamaguchi, *Materials Transactions, JIM.* **7**, 918 (1995).
7. A. Weidenmann, *J. Appl. Cryst.* **33**, 428 (2000).
8. U. Keiderling and A. Weidenmann. *Physica B* **213-214**, 895 (1995).
9. K. Handrich, *Phys. Stat Solidi* **32**, K55 (1969).
10. A. Guinier and G. Fournét, *Small-Angle Scattering of X-Rays*, Wiley, New York (1955).
11. A. Heinemann, H. Hermann, A. Weidenmann, N. Mattern, K. Wetzig, *J. Appl. Cryst.* **33**, *J. Appl. Cryst.* **33**, 1386 (2000).